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1-फिनाइल-3-मिथाइल-5-पायराज़ोलोन —  
विशिष्टि  
( पहला पुनरीक्षण )

1-Phenyl-3-Methyl-5-Pyrazolone —  
Specification  
( First Revision )

ICS 71.080.99

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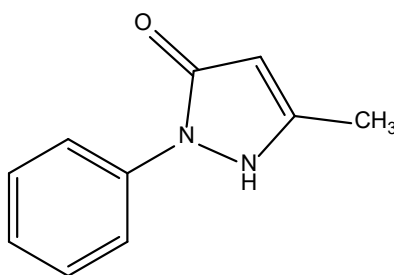


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## FOREWORD

This Indian Standard (First Revision) is adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee has been approved by the Petroleum, Coal and Related Products Division Council.

1-Phenyl-3-methyl-5-pyrazolone ( $C_{10}H_{10}N_2O$ ), is an important dye intermediate used in the manufacturing of azo dyes. It has the following structural formula:



1-Phenyl-3-methyl-5-pyrazolone  
(Molecular mass: 174.2)  
CAS Number – 89-25-8

1-Phenyl-3-methyl-5-pyrazolone is harmful to aquatic life with long lasting consequences. Therefore, release of 1-phenyl-3-methyl-5-pyrazolone in environment should be prevented. Further, contents/containers/bags are disposed of in accordance with national regulations. This standard was first published in 1977.

In this revision, determination of assay by nitrite value and by HPLC have been incorporated. Solubility in sodium hydroxide solution is substituted by matter insoluble in sodium hydroxide solution with the limit of 0.50 percent by mass, maximum.

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement is given in Annex C, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***1-PHENYL-3-METHYL-5-PYRAZOLONE — SPECIFICATION***( First Revision )***1 SCOPE**

This standard prescribes the requirements, the methods of sampling and test for 1-phenyl-3-methyl-5-pyrazolone.

**2 REFERENCES**

The standards given below contain provisions which through reference in the text constitute provisions of this standard, at the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 5299 : 2001	Methods of sampling and tests for dye intermediates ( <i>first revision</i> )
IS 5762 : 1970	Methods for determination of melting point and melting range
IS 14887 : 2014	Textiles — High density polyethylene (HDPE)/polypropylene (PP) woven sacks for packaging of 50 kg food grains — Specification ( <i>first revision</i> )

**3 REQUIREMENTS****3.1 Description**

The material shall be in the form of pale-yellow crystals and shall be free from visible impurities.

**3.2** The material shall also comply with the requirements as given in Table 1, when tested according to the methods prescribed col (4) and (5) of Table 1.

**4 PACKING AND MARKING****4.1 Packing**

The material shall be packed in HDPE/PP woven sacks

(see IS 14887). Each bag shall be securely closed.

**4.2 Marking**

**4.2.1** Each bag shall bear legibly and indelibly the following information:

- Name of the Material;
- Name of the manufacturer/supplier, complete address and his recognized trade-mark, if any;
- Gross, net and tare mass;
- Batch number, month and year of manufacturing;
- Shelf life of the material; and
- Any other statutory requirement.

**4.2.2 BIS Certification Marking**

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

**5 SAMPLING**

**5.1** The method of drawing representative samples of the material shall be as prescribed in 4 of IS 5299.

**5.2 Number of Tests**

**5.2.1** Test for assay by nitrite value shall be conducted on each of the individual samples.

**5.2.2** Tests for determination of all other characteristics namely, assay by HPLC, melting point and matter insoluble in sodium hydroxide solution, given under Table 1, shall be conducted on the composite sample.

**5.3 Criteria for Conformity****5.3.1 For Individual Samples**

The lot shall be declared as conforming to the requirement of assay by nitrite value if each of the individual test result satisfies the relevant requirements given in Table 1.

**5.3.2 For Composite Samples**

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements in Table 1.

**6 TEST METHODS****6.1 Preparation of sample**

Dry the material at  $55\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  to constant mass. Grind and mix well. Transfer the material to a wide-

mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

**6.2** Test shall be carried out according to the methods prescribed in col (4) and (5) of Table 1.

**6.3 Quality of Reagents**

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**Table 1 Requirement for 1-Phenyl-3-methyl-5-pyrazolone**

(Clauses 3.2, 5.2.2, 5.3.1, 5.3.2 and 6.2)

SI No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay (by nitrite value), percent by mass (on dry basis), <i>Min</i> or	97.0	A	—
ii)	Assay (by HPLC <sup>1)</sup> ), percent area (on dry basis), <i>Min</i>	99.0	B	—
iii)	Melting point, $^{\circ}\text{C}$	127 to 129	-	IS 5762
iv)	Matter insoluble in sodium hydroxide solution, percent by mass, <i>Max</i>	0.50	-	11.2 of IS 5299

<sup>1)</sup>In case of disputes, determination of assay by HPLC, shall be the referee method.

## ANNEX A

[Table 1, Sl No.(i)]

## DETERMINATION OF 1-PHENYL-3-METHYL-5-PYRAZOLONE CONTENT (ASSAY) BY NITRITE VALUE

## A-1 REAGENTS

## A-1.1 Concentrated Hydrochloric Acid

## A-1.2 Potassium Bromide

## A-1.3 Standard Sodium Nitrite Solution — 0.1 N

## A-1.4 Potassium Starch Iodide Indicator Paper

## A-1.5 Ice

## A-2 PROCEDURE

Weight 8 g to 10 g dry powder in 250 ml glass beaker. Add distilled water approximately 150 ml and stir with glass rod to make a smooth slurry. Add 20 percent soda ash solution (approximately 7 ml to 10 ml) to dissolve the powder and make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 ml of the solution by using pipette in to 1 000 ml beaker. Add 200 ml to 250 ml distilled water. Add ice cubes to make the temperature around 10 °C.

Weigh and add 1 g potassium bromide into the cold solution. Add hydrochloric acid to make the pH acidic (pH around 2 to 2.5 on pH paper) approximately 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the endpoint to put the spot-on starch iodide paper, the end-point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

## A-3 CALCULATION

Assay (by nitrite value), percent by mass (on dry

$$\text{basis}) = \frac{V \times N \times 174.2}{M}$$

where

$V$  = volume, in ml, of standard sodium nitrite solution used in the titration.

$N$  = normality of sodium nitrite solution; and

$M$  = Mass, in g, of the dry material taken for the test,

## ANNEX B

[Table 1, Sl No. (ii)]

## DETERMINATION OF 1-PHENYL-3-METHYL-5-PYRAZOLONE CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

## B-1 OUTLINE OF METHOD

High performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

## B-2 APPARATUS

**B-2.1 HPLC** — isocratic liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

**B-2.1.1 Column** — C18 column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

## B-2.2 Analytical Balance

## B-3 REAGENT

**B-3.1 Tetrabutylammonium Hydrogen Sulphate** — HPLC grade

**B-3.2 Dipotassium Hydrogen Phosphate** — analytical grade (AR)

**B-3.3 Acetonitrile** — HPLC grade

**B-3.4 Methanol** — HPLC grade

**B-3.5 Water** — HPLC grade

**B-3.6 1-Phenyl-3-Methyl-5-Pyrazolone** — known purity

#### B-4 SAMPLE PREPARATION

Weigh accurately 0.010 g (10 mg) of dry 1-phenyl-3-methyl-5-pyrazolone in 100ml volumetric flask. Dissolve it in water and make it up to 100 ml with water.

#### B-5 BUFFER PREPARATION

Take 12 g tetrabutylammonium hydrogen sulphate (TBAHS) and 29 g dipotassium hydrogen phosphate ( $K_2HPO_4$ ) in 190 ml water and dissolve (stock solution).

**B-6 FLOW RATE** — 1 ml/min

**B-7 MOBILE PHASE** — 100 ml water + 4 ml buffer + 70 ml acetonitrile + 7 ml methanol

**B-8 COLUMN OVEN TEMPERATURE** — 26 °C

**B-9 INJECTION VOLUME** — 20 µl

**B-10 RUN TIME** — 10 min, *Max*

**B-11 WAVELENGTH** — 254 nm

#### B-12 PEAK TIME

#### B-13 CALCULATION

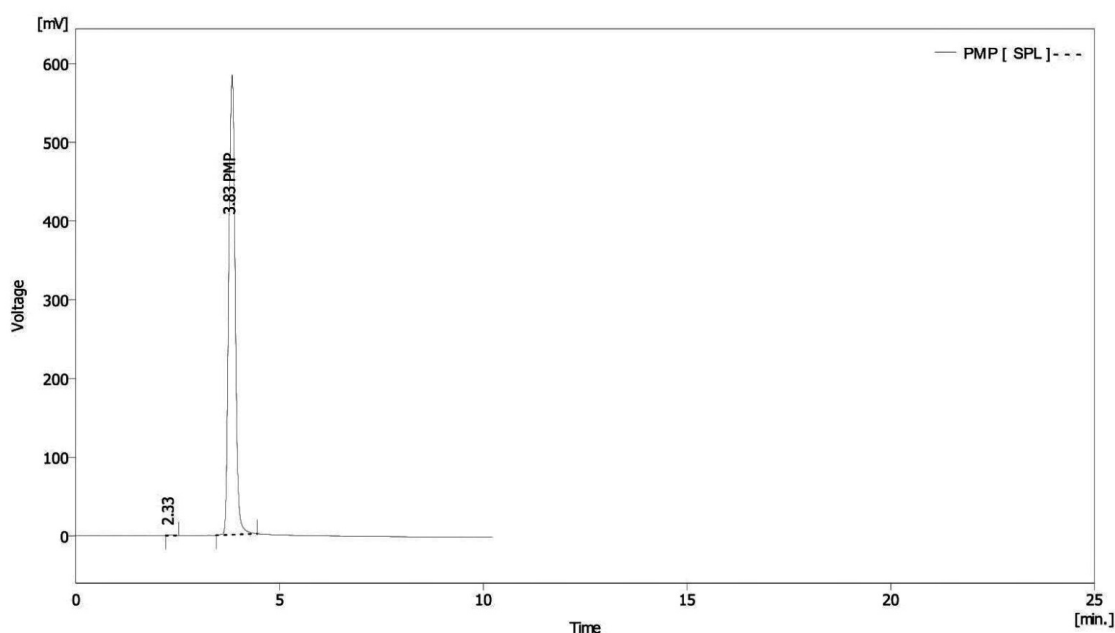
Calculate the peak area of individual constituent pertaining to 1-phenyl-3-methyl-5-pyrazolone from the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure 1-phenyl-3-methyl-5-pyrazolone.

1-Phenyl-3-methyl-5-pyrazolone, percent =

$$\frac{A}{\text{Total Area}} \times 100$$

where

A = area of peak 1-phenyl-3-methyl-5-pyrazolone in sample.




1-Phenyl-3-methyl-5-pyrazolone: 3.8 min

FIG. 1 TYPICAL CHROMATOGRAM

## ANNEX C

(Foreword)

## PICTOGRAMS, SIGNAL WORD, HAZARD STATEMENT AND PRECAUTIONARY STATEMENT

<b>Pictogram(s)</b>	:	
<b>Signal word</b>	:	<b>WARNING</b>
<b>Hazard statement(s)</b>	:	<p>H317: May cause an allergic skin reaction.</p> <p>H412: Harmful to aquatic life with long lasting effects.</p>
<b>Precautionary statement(s)</b>	:	<p><b>Prevention:</b></p> <p>P261: Avoid breathing dust/fume/gas/mist/vapours/spray.</p> <p>P272: Contaminated work clothing should not be allowed out of the workplace.</p> <p>P273: Avoid release to the environment.</p> <p>P280: Wear protective gloves/protective clothing/eye protection/face protection.</p> <p><b>Response:</b></p> <p>P363: Wash contaminated clothing before reuse.</p> <p>P302 + P352: IF ON SKIN: Wash with plenty of soap and water.</p> <p>P333 + P313: If skin irritation or rash occurs. Get medical advice/attention.</p> <p><b>Disposal:</b></p> <p>P501: Dispose of contents/container in accordance with local/regional/national/international regulations.</p> <p>Waste treatment in accordance with national regulations.</p>

**ANNEX D***(Foreword)***COMMITTEE COMPOSITION**

Dye Intermediates Sectional Committee, PCD 26

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### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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